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A. A. BRENEMAN, 97 Water St., New York.

ON A NEW METHOD OF ESTIMATING COLORING MATTER IN BUTTER AND ITS SUBSTITUTES.

BY JAMES H. STEBBINS, JR.

Butter varies in color, according to the season of the year in which it is made.

In summer, when the cows are fed upon grass, it is much more yellow than in winter, when the cows are fed upon dry fodder. In order, therefore, to render butter of as uniform a color as possible throughout the whole year, dairymen and farmers have been accustomed to add, or rather incorporate with the butter, a certain amount of coloring matter. This is done, not only for the above reason, but also to cater to the taste of people who think that a highly colored butter is much richer and of better quality than one not so highly colored. It is useless here to dwell upon the absurdity of this idea. Several dyestuffs have been used to color butter, but those with which we have mostly to deal are annatto, turmeric, saffron and carrotine.

In order to determine the nature of the coloring matter which may have been added, it has been customary to extract the fat with weak boiling alcohol, and to make tests upon the solution so obtained. This method I have found to be very unsatisfactory for two reasons: 1st. Because it is very difficult to thoroughly extract coloring matter from the fat, on account of the insolubility of the latter in boiling alcohol. 2d. Because the small amount of fat which is dissolved by the alcohol impairs the sharpness of the color tests with sulphuric acid. These tests are therefore only applicable to alcoholic extracts of coloring matter containing no fat in solution.

Some time ago (*Analyst, 10*) Mr. Martin devised a method for determining some of the coloring matters in butter. In carrying out this test he dissolves a small quantity of fat (5 grms.) in 20 c.c. of carbon disulphide, and adds thereto 25 c.c. of solution of KOH, containing 2 drops of normal caustic potash. The mixture is now thoroughly shaken and allowed to stand. The coloring matter is said to pass into the aqueous solution, and may then be drawn off and further examined.

I have tried this test repeatedly, and have found that the aqueous solution becomes colored, whether the butter under examination had been colored or not. Furthermore, I have made a number of blank tests with both perfectly pure CS_2 and the commercial article, and have found that even here the aqueous alkaline solution is colored more or less yellow, according to the length of time during which the dilute potash solution is allowed to react upon the CS_2. The formation of the above mentioned brownish yellow color is really due to a chemical reaction between the CS_2 and KOH.

$$CS_2 + 2\,KOH \quad CS\begin{matrix}SK\\OK\end{matrix} + H_2O$$

It is therefore evident that this test is misleading.

Mr. Martin has now perfected another method, which, briefly described, is as follows:

A small amount of butter fat, freed from water, curd and salt, is dissolved in a known quantity of CS_2 and to this solution is added an equivalent quantity of methyl alcohol. The mixture is now thoroughly shaken and then allowed to stand until complete separation of CS_2 and methyl alcohol has taken place. It will be found that more or less coloring matter will have passed into the alcohol, which may then be decanted and further examined.

This test is an improvement on his first one, but is still defective, for two reasons: 1st, because a small amount of fat is liable to be dissolved by the alcohol, and interfere with the sulphuric acid test; and 2d, because by this means it is almost impossible to eliminate any carrotine which might be present. Carrotine is easily soluble in CS_2 and very difficultly soluble in alcohol. It is therefore evident that the carrotine, if present, will remain dissolved in the CS_2 solution, and that any amount of shaking with methyl alcohol will fail to remove it.

In the method to be described, I think that all of the above mentioned objections have been successfully overcome.

The test is based upon the observation made by me about a year ago, that if finely pulverized fullers' earth be added under constant stirring to hot, melted butter fat, and the mixture be kept warm until all the earth has settled to the bottom of the beaker, all of the coloring matter will be carried down with the fullers' earth.

The test is carried out as follows:

50 grms. of butter fat, freed from curd, salt and water, are weighed out into a small narrow beaker. The fat is then melted on a steam bath, and into it is stirred 5 or 10 grms. of finely pulverized fullers' earth. The mixture should now be thoroughly stirred for two or three minutes and then allowed to remain upon the steam bath until the earth has fully settled. As much of the clear fat as possible is now poured off into another beaker, without disturbing the sediment. To the sediment remaining in the first beaker is added 20 c.c. of benzole; the mixture is thoroughly stirred with a glass rod, the earth allowed to settle, and the benzole poured off into a clean filter.

This washing with benzole is repeated until a few drops, evaporated upon a watch glass, leave no fatty residue.

The filter paper is now rinsed with a little more benzole, and all the filtrates united in a flask. If carrotine had been used for coloring the butter, it will be contained in the benzole, and may be tested for in the usual manner. Carrotine is very easily soluable in C_6H_6, and very difficultly soluble in alcohol. There will, therefore, be no trouble in totally extracting it from the earthy sediment. The earthy sediment, freed from fat and carrotine, is now heated upon the water bath, until the last traces of benzole have been driven off. It is then boiled out three times with about 20 c.c. of 94% alcohol. The alcoholic extracts are filtered into a tared evaporating dish, and the alcohol is then driven off. The residue left in the dish, composed of curcumine or annatto, or possibly both, is dried to constant weight at 100° C., weighed, and then tested colorimetrically in the usual manner.

Curcumine and annatto are nearly insoluble in benzole, but are, on the contrary, very easily soluble in alcohol. It is therefore not likely that any of these coloring matters will be extracted by the benzole in the first operation.

DRINKING WATER AND DISEASE.

BY PROF. W. P. MASON.

In view of Dr. Emmerich's experiments as to the effect of drinking the diluted sewage from healthy persons, there would seem to be small danger lurking in excremental material known to come from a non-pathological source.

An interesting case has recently come under my observation, which shows conclusively that even normal sewage may not be taken inwardly with impunity. Appended is the analysis of a cistern water from Albany, N. Y. The cistern is of the "filtering" type—the filtering wall being of some thickness. The property is owned and occupied by wealthy persons, and every care has been exercised, presumably, in keeping the premises in good order.

Suspicion was cast upon the water some months since, but its use was continued under a belief in the cleanliness of the cistern. Suddenly, with but a few hours warning, the entire household, one person excepted, were taken ill. The symptoms varied from intense nausea to great, if not dangerous, prostration. Over a month has now elapsed, yet the effects are still felt in a measure by those more severely attacked. The one person who escaped was the one who did not drink the water. Among those attacked were several children.

Upon investigation, it was found that both cistern lining and private sewer were defective, and that more or less direct communication existed between them. Up to the time of this outbreak, none of the household had suffered from diarrhœa or kindred troubles, nor could I obtain any history of recent disease of any kind. The children attacked were on a short visit only.

What I particularly desire to point out is, that the sewage, which undoubtedly caused the trouble, was *normal;* probably not *fresh,* but still *normal.* The absence of nitrates would go in part, moreover, to show that it was not very old.

To my mind, the evidence seems pretty clear that "specific" germs are not essential to a sewage, in order to make its presence in drinking water strongly objectionable. It is greatly to be regretted that previous analyses of the water were not made, as thereby it is possible some information might have been obtained

as to the relation of the figures of the analysis to the "poisoning point" of the water.

ANALYSIS:

	Parts per million.
Free Ammonia	15.000 ! ! !
Albuminoid Ammonia	2.800
Chlorine	46.000
Nitrites and Nitrates	traces.

ACTION OF OFFICINAL NITRIC ACID ON SUGAR.

BY PROF. W. P. MASON.

As every one knows, strong nitric acid acts upon sugar with production of oxalic acid.

Let us represent the reaction by the equation:

$C_{12}H_2\ O_{11} + 36\ HNO_3 = 6\ H_2C_2O_4 + 18\ N_2O_4 + 23\ H_2O.$

Officinal nitric acid has a specific gravity of 1.068 corresponding to 11.66 % pure HNO_3. One hundred c.c. of such acid, acting upon sugar during a period of two days, gave no trace of oxalic acid at the end of that time. Fifteen drops of officinal acid, further diluted with water, is the maximum dose. As was to have been expected, this still weaker solution was also without action on the sugar during the stated two days time.

I mention this fact simply because it has recently come to my knowledge that a physician of this vicinity was discharged on the ground of ignorantly poisoning his patient with oxalic acid, by advising the sweetening of an ordinary dose of nitric acid.

ABSTRACTS.

ANALYTICAL CHEMISTRY.

A New Method for the Determination of Starch. A. Von Asboth.

The direct determination of starch in cereals, by inverting the starch with hydrochloric acid and determining the resulting sugar by means of Fehling's solution, is subject to serious errors, inasmuch as some of the celluiose is liable to be changed into sugar, and also that some of the sugar resulting by inversion is further decomposed by the prolonged boiling.

The author, acting upon the statement of Zulkowsky (*Wagner, Jahresb. 1878, p. 753*) that baryta water produces insoluble precipitates in solutions containing soluble starch, found on investigation that the precipitate is of uniform composition, having the formula $BaOC_{24}H_{40}O_{20}$ and containing 19.10% of BaO. The precipitate is slightly soluble in water, but insoluble in excess of baryta water or alcohol.

Forty-five per cent. alcohol completely separates the precipitate from dilute solutions.

For starches the following method was used: About 1 grm. starch is digested on the water-bath, in a 250 c.c. flask containing 150 c.c. of water, until the starch is changed to a clear paste. 50 c.c. of baryta water are then added to the warm solution, the flask securely stoppered and the mixture well shaken for about two minutes, and finally 45% alcohol is added to dilute the mixture to 250 c.c. It is allowed to cool, filled up to the mark with 45% alcohol, and, after again thoroughly mixing, the precipitate is allowed to separate. In the meantime 10 c.c. of the baryta water are titrated with $\frac{N}{10}$ hydrochloric acid, using phenolphthalein as indicator, and deducting the number of c.c. of $\frac{N}{10}$ acid used, from the amount required to neutralize 50 c.c. of the clear liquid from the flask. The starch and baryta water mixture generally clarifies within ten minutes. The difference between the two titrations multiplied by 5 and then by 0.0324 (the starch equiv. of 1 Ba O) gives the weight of starch in grammes. It is necessary to use well boiled distilled water in the above work and to supply

the baryta water apart from contact with the air, so as to prevent precipitation of carbonate.

For determining the starch in cereals, the following modification of the process was used. About 3 grms. of the cereal is rubbed up in a mortar with very little water, and by successive washings the starch gradually transferred to a 250 c.c. flask. Finally, the residuum is also transferred to the flask and the whole diluted to about 100 c.c.

For very hard substances, such as corn, rice, peas, etc., hot water may be used as above. Then place the whole for 30 minutes into boiling water until the starch is dissolved, cool and add 50 c.c. baryta water and shake the mixture for about 2 minutes. Fill up to mark with 45% alcohol, mix and let the mixture stand for about 10 minutes. If necessary filter a portion of the clarified mixture through glass, wool or asbestos. Titrate 50 c.c. and proceed as before.

In examining various cereals and using the above method for the starch determinations, the author apparently shows that the cereals contain little or no sugar, whereas, by use of other methods, often 4% of sugars have been found. In peas the author found that the filtrate from the baryta precipitate obtained as above, contained a substance which, besides giving the various protein reactions, also, after inversion by hydrochloric acid, reduced alkaline copper solution. (*Rep. d. Anal. Chem.* **8**, *299–308.*)

J. F. G.

Determination of Albumen according to Milton and Commaille. R. Palin.

In the precipitation of albumen by mercuric nitrate or acetate all the albumen is precipitated, but the acids simultaneously liberated dissolve some of the precipitate formed, and therefore cause a loss. A slight excess of dilute potassium hydrate solution added gradually to neutralize the liberated acid which is set free during the precipitation will cause the albumen to be completely precipitated.

1. The precipitate is collected on a tared filter, thoroughly washed with water, dried at 100° C. and weighed. The mercuric oxide is then determined and deducted from the first weight, the difference being the amount of albumen.

2. Or, the precipitate caused by the mercuric nitrate is thoroughly washed and then digested with a solution of barium hydrate, filtered while hot, and after cooling the barium is precipitated by neutralizing with dilute sulphuric acid. It is filtered to remove the barium sulphate, the filtrates evaporated, dried at 100° C. and weighed, giving the weight of protein. (*Fres. Zeit.* **26,** *329.*)

J. F. G.

(N. B.—It is well to ignite the last residue and deduct the resulting ash. J. F. G.)

Determination of Protein Compounds in Milk. R. PALM.

The author, as a result of a comparison of the different methods for determining the protein compounds of milk, considers the precipitation of the proteids by lead oxide as the only method giving correct results, for by the ordinary methods the hemialbumen and milk peptone are not precipitated and therefore lost. The following method was therefore used: To 10 g. of milk 0.2-0.3 g. of lead oxide (litharge) are added and the whole evaporated, by which the protein compounds combine with the lead oxide. The residue is then washed with water and transferred to a tared filter. After thorough washing, it is dried at 100° C. and weighed, which gives the weight of proteins plus lead oxide. The latter is then determined by ignition of the residue, and deducted from the weight of protein and lead oxide, the difference being the protein in the quantity of milk taken.

If it is desired to make a quantitative separation of the different protein compounds the above method can be applied to the filtrates after the separation of the albumen and casein. (*Fres. Zeit.*, **26,** *319–329.*)

J. F. G.

Abstracts of American Patents Relating to Chemistry.

(*From the Official Gazette of the U. S. Patent Office.*)

February 15th, 1887.

357,659.—Obtaining chlorine by electrolysis. D. G. Fitzgerald.

Chlorine is generated by means of an anode of lead peroxide, in conjunction with any suitable cathode, and with an electrolyte capable of evolving chlorine.

357,696.—Carbonic acid gas generator. J. F. Behlmar.

February 22d, 1887.

358,082.—Composition of matter as a substitute for hard rubber. A. W. Sperry.

Composed of ivory dust or the like material, making up substantially fifty per cent. of the compound, a starch mixture consisting of starch, tannin, and an alkali, as alum, a binder mixture consisting of a resinous gum dissolved in alkali, as caustic soda, and milk, glue, shellac and alcohol, the whole combined and intimately mixed.

358,196.—Apparatus for distilling ammonia. H. Grüneberg and E. Blum.

358,213.—Process of preserving milk. J. Meyenberg.

358,300.—Manufacture of liquid extracts of coffee. M. Samelson.

The extract is prepared by first treating coffee with cold water, then with warm water, and finally with boiling water, and combining the three fluids.

March 1st, 1887.

358,612.—Baking powder. A. W. Holway.

Consists of mucic acid, an alkaline carbonate, and anhydrous starch.

358,662.—Process of producing silicious metals and utilizing the same for metallurgical purposes. C. Hensler.

Silicious copper, silicious tin, and silicious zinc are produced by melting the respective metals with silicious iron.

358,816.—Photography. A. M. Jacobs.

358,817.—Photographic reproduction. A. M. Jacobs.

March 8th, 1887.

358,865.—Production of mixed azo coloring matter. C. A. Martins.

358,901.—Fluid ink eraser. J. W. Tallmadge.

Consists of acetic acid and solution of calcium hypochlorite.

359,148.—Separation of glycerin from fats. R. Giebermann.

W. R.

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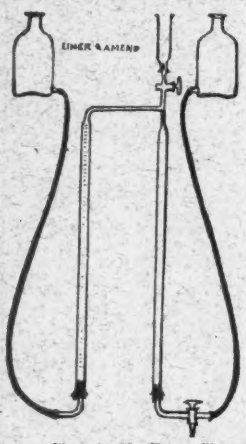

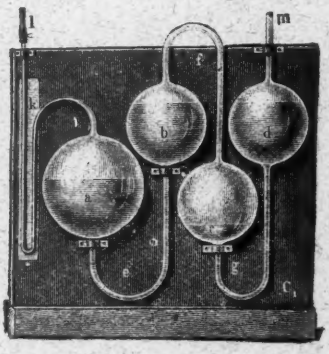

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